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METALLURGICAL STUDY OF CRITERIA USED TO ACHIEVE COMPRESSION OF ELEVATED TEMPERATURE TEST TIME

R. E. Herfert

Northrop Corporation

TECHNICAL REPORT AFML-TR-70-57

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FOREWORD

This final report was prepared by the Materials Research Department, Northrop Corporation, Aircraft Division, under USAF Contract No. F33615-69-C-1540. This contract was initiated under Project Number 7351. This work was monitored by Dr. W. H. Reimann of the Air Force Materials Laboratory. The manuscript was released by the author for publication.

This report covers work conducted in the period from 1 April 1969 to 10 April 1970. Report Number NOR-70-20 has been assigned for internal control.

Personnel participating in the work included L. Stone, G. Blake, R. Rosas, J. Donner, and J. Abger.

This technical report has been reviewed and is approved.

W. J. Trapp

W. J. Trapp
Chief, Strength and Metals Dynamic Branch
Ceramics Division
Air Force Materials Laboratory

ABSTRACT

This investigation was performed to study the validity of certain criteria used to achieve compression of test time of elevated temperature creep tests. The hypothesis that accumulation of a constant strain under various stresses at a constant temperature results in equivalent damage was evaluated from residual strength level as well as microstructural behavior. Materials selected for this evaluation were Ti-8Al-1Mo-1V in the duplex-annealed condition, Ti-6Al-4V in the annealed condition, and aluminum alloy 2024-T3. Materials were chosen as representative of high strength titanium alloys and aluminum alloys having good elevated-temperature strength. The titanium alloys were creep strained to 1% total strain using three different creep stresses at 800F; the aluminum alloy was investigated using a similar approach at 300F. Detailed microscopic studies were performed to study microstructural changes in terms of the creep rate. Residual strength was correlated with microstructure to determine the validity of the "equivalent damage" approach to test time compression.

It was determined that the hypothesis of a constant strain resulting in equivalent damage was not universally valid. Metallurgical changes as a result of thermal exposure and creep straining resulted in changes in tensile strength behavior. Examination of the microstructure could be directly correlated with mechanical behavior changes.

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INTRODUCTION

With the inception of aircraft which are designed for service life up to 50,000 hours, a need for knowledge of the behavior of materials in service exposure is required. With the supersonic vehicles, an elevated temperature requirement is present. It is not practical to run real life tests due to the length of time (50,000 hours ~ 6 years). Methods are being investigated for accelerating these tests.

Elevated temperature tests under steady and/or alternating stress can only be accelerated by increasing stress, temperature, or loading frequency. However, the same changes in microstructure should occur in an accelerated test as would take place in a real-time test. Because different metallurgical changes may occur in primary creep as compared to secondary or tertiary creep, one would not attempt to accelerate a creep test by a change from one region of creep to a different region. The two specimens will probably not undergo equivalent microstructural changes. In this study, some initial aspects of compressing elevated temperature creep tests were investigated. The effect of accumulating a constant creep strain at different rates was studied in terms of metallurgical changes and residual strength.

In the investigation "Study to Determine the Suitability of Compressing the Time of Mission Profile During Elevated Temperature Fatigue Testing on Large or Full-Scale Vehicles," Contract Number AF33 (657)-11323[1,2], a method is proposed for test-time compression involving the use of a constant creep strain accumulation as a criterion for obtaining equivalent damage during an accelerated elevated temperature fatigue test involving prior creep. This method essentially states that regardless of the amount of time or the temperature to obtain a specific strain level, the same amount of damage has been performed on the material under test.

This program was designed to determine whether this hypothesis, accumulation of a constant strain under various stresses at a constant temperature, results in equivalent damage in the alloys Ti-8Al-1Mo-1V, Ti-6Al-4V, and 2024-T3.

EXPERIMENTAL PROCEDURES

Materials

Ti-6Al-4V in the annealed condition and Ti-8Al-1Mo-1V in the duplex-annealed condition were obtained as sheets of nominally 0.063-inch thickness, 48-inch by 96-inch. The Ti-6Al-4V was ordered under Specification AMS-4911, and the Ti-8Al-1Mo-1V under MIL-T-9046, Type 2, Comp. F. Sufficient material was ordered not only to complete the tensile behavior program but to have sufficient material for subsequent studies of fatigue behavior. Since a sheet of 2024-T3 of 0.160-inch thickness for which creep data had already been obtained was available, this material was used for studies in this program. Table I shows the chemical analyses of all three starting materials.

A specimen design was adopted which would easily lend itself to creep straining, tensile testing, and subsequent fatigue testing. A drawing of the specimen used in this program is shown in Figure 1. Tensile tests were performed at room temperature and 800F for the Ti-8Al-1Mo-1V and Ti-6Al-4V alloys and at room temperature and 300F for the 2024-T3 aluminum alloy. Yield tensile strength (YTS), ultimate tensile strength (UTS), and elongation were measured for each specimen. A minimum of three specimens of each material were run at each temperature. The tensile properties of the as-received materials are shown in Table II.

Thermal Exposure

In order to determine the thermal stability of these three alloys without the effects of strain accumulation, twelve tensile coupons of each alloy --- 2024-T3, Ti-8Al-1Mo-1V, and Ti-6Al-4V --- were exposed at elevated temperatures, four specimens at each of three times, 50, 250, and 500 hours. The titanium alloys were exposed at 800F and the 2024-T3 alloy at 300F in a recirculating air furnace. Temperature control was monitored by thermocouples attached to bundles of tensile coupons. Temperature variation was found to be within ± 5F.

Creep Straining

In order to select suitable stresses for the accumulation of 1% strain in each of the three alloys under study, constant strain diagrams were established by running several static creep tests to above 1% total strain or rupture. A creep strain, here defined as the total creep strain excluding the instantaneous strain on loading, of 1% was selected for this program.

Three Satec Model 12 Creep Machines and a Riehle CR-12 Creep Machine, all with 20,000-pound capacity, were used for all creep straining and testing. Kanthal-wound furnaces with three-zone temperature controls were used on all creep machines. Temperature control was better than ± 3F from the top of the gage length to the bottom of the gage length. Static creep tests were not run on the Ti-8Al-1Mo-1V

alloy since voluminous amounts of data were available from the AFFDL program [1,2]. It was only necessary to verify by a few tests that the Ti-8Al-1Mo-1V material for this program was equivalent to that used on the AFFDL program[1,2].

Model 200 extensometers equipped with a linearly variable differential transformer were used for recording strain during testing. Both knife grip and point contact extensometer heads were used for strain measurement. Difficulties were encountered using the point contact extensometer head on the 2024-T3. It was possible to create premature failure by over-torqueing the extensometer head. A study of failure time versus torque was run on spare specimens of aluminum in order to eliminate premature failures. No difficulties were encountered with the knife edge extensometer heads. The extensometer heads were placed at a distance of 0.90-inches about the center of the gage length rather than the conventional 1.00-inch. Recorders were set to show full-scale deflection as 0.1-inch extension. Additional measurement of total strain was achieved by lightly scribing marks 1.00-inches about the center of the gage length. This distance was accurately measured prior to creep straining using a traveling microscope. Total strain accumulation was then recorded using the same traveling microscope after straining. Correlation between extensometer and traveling microscope strain measurements were exceptionally good. Comparison of both methods showed agreement to be within ± 3% of each other.

Microstructural Studies

As-received materials, 50 and 500-hour thermal exposure samples, and 50 and 500-hour 1% strain specimens were examined using optical and electron metallography and transmission electron microscopy. Metallographic specimens were removed from the center of the gage length area and mounted to show both the transverse and longitudinal direction of the straining. Optical examination was performed at magnifications of 500 and 1000X. Specimens were lightly etched, 2024-T3 with Keller's reagent, and Ti-8Al-1Mo-1V and Ti-6Al-4V by Kroll's etchant. Two-stage carbon-chromium replicas were prepared from the metallographic samples. Electron metallographic examination was performed at 3000X, 5000X, and 10,000X.

Samples for transmission electron microscopy of 3/8-inch by 1/2-inch by 0.005-inch were sliced from the center of the gage length area of the tensile coupon using a Brownwill Scientific thin sectioning machine. The thin foils of titanium were prepared in two steps. First, a bath of 30% hydrofluoric acid and 70% nitric acid diluted with 50% of water was used to chem-mill the sample to 0.002-inch. The sample was then transferred to a Precision Scientific dual-jet polisher/thinner. The Disa A-3 Electrolyte was used for final electropolishing of the foils of titanium to a thickness of approximately 2,000Å. The samples of 2024-T3 were sectioned similarly. Chem-milling was not performed on this material. The Disa A-2 Electrolyte was used to thin the 0.005-inch slices directly to approximately 2,000Å. Examination was performed at 10,000X. Identification of precipitates and of the orientation of foils was determined through the use of selected area electron diffraction.

In the instances where fractographic examination was performed on tensile coupons, a Jeolco JSM-2 scanning electron microscope was used for examination. Representative photographs were taken at 1,000X and 3,000X.

EXPERIMENTAL RESULTS

Establishment of Constant Strain Diagrams

In order to select suitable stresses to obtain 1% strain in 50, 250, and 500 hours, it was first necessary to construct constant strain diagrams for 2024-T3 at 300F and Ti-8Al-1Mo-1V and Ti-6Al-4V at 800F. The creep strain used here is the total creep strain, excluding the time-independent plastic strain that occurs on initial loading. Data for the Ti-8Al-1Mo-1V alloy at 800F was obtained from the Air Force Flight Dynamics Laboratory program, "Study to Determine the Suitability of Compressing the Time of Mission Profile During Elevated Temperature Fatigue Testing of Large or Full-Scale Vehicles"[1,2]. Since this data was obtained on the material used in the referenced program, it was necessary to verify the similarity of behavior of the material obtained for this study by running a few additional creep rupture tests. Creep rupture tests were made on the 2024-T3 and Ti-6Al-4V at a sufficient number of loads to obtain a cross plot of data for stress to produce 1% creep strain as a function of time and temperature. Strain versus time curves for 2024-T3 at 300F were obtained for stresses of 20 ksi, 45 ksi, 50 ksi, and 55 ksi. Similarly, curves were obtained for the Ti-6Al-4V at 800F for stresses of 60 ksi, 65 ksi, 72 ksi, and 75 ksi. Creep rupture tests on the Ti-8Al-1Mo-1V were performed at 74 ksi and 70 ksi. Tables III, IV, and V show a tabulation of the creep rupture and prestraining tests performed.

No problems were encountered with the creep testing of the two titanium alloys. Difficulty was encountered with the 2024-T3 alloy in that aging resulting in embrittlement was obtained for creep exposures between 400 and 500 hours. It was not possible to run a 500-hour test at a load selected to obtain 1% strain. However, specimens could be strained to approximately 1% strain in 400-440 hours. The program was run with concern being placed on the obtaining of 1% strain rather than stopping tests at a fixed time of 50, 250, or 500 hours.

Constant strain diagrams are plotted in Figure 2. A compilation of the strain and time values in Table III shows a close correlation of strain with a constant stress (note 51 ksi data) for the 2024-T3 alloy. However, the constant stress (68 ksi) applied to the Ti-6Al-4V showed a spread of 1.2% to 2.1% strain for 38-39 hours. This appeared indicative of the spread in material properties for titanium on a sample-to-sample basis.

Thermal Exposure Studies

Specimens machined to the configuration in Figure 1 were exposed to a temperature of 300F for the 2024-T3 alloy and 800F for the titanium alloys for periods of times of 50, 250, and 500 hours. These specimens were then tensile tested. The tensile properties of the thermally exposed samples are shown in Table VI. This data is graphically represented in Figures 3, 4, and 5.

Creep Straining Results

Specimens strained nominally to 1% strain at elevated temperatures (300F for the 2024-T3 and 800F for the Ti-8Al-1Mo-1V and Ti-6Al-4V) were tensile tested at room temperature and elevated temperature (300F or 800F). Tables III, IV, and V show the complete compilation of creep strain tests and tensile properties obtained at ambient and elevated temperatures. Comparison of the tensile results from the thermal exposure and strained specimens for all materials are shown in Figures 6, 7, 8, 9, 10, and 11.

Microstructural Studies

As-Received Materials - Examinations by optical and electron metallography and transmission electron microscopy (TEM) were performed on each starting material. Representative photomicrographs were taken at 500X, 1000X, 3000X, and 10,000X. Two-stage carbon-chromium replicas were used for electron metallographic purposes. Representative high magnification photomicrographs of each material are shown in Figure 12. The microstructure of the 2024-T3 showed an elongated grain structure parallel to the rolling direction with particles primarily of $CuAl_2Mg$. The $CuAl_2$ particles were undissolved constituents carried through the solution treatment process. The Ti-6Al-4V showed the typical equiaxed alpha grain structure with beta particles located at the grain boundaries. The Ti-8Al-1Mo-1V microstructure showed the two types of alpha, equiaxed and aged (striated), plus beta particles at the grain boundaries.

Transmission electron microscopy of the 2024-T3 condition showed, in addition to the large $CuAl_2$ particles, small spherical G.P. zones as a result of the room temperature aging process (Figure 13). TEM examination of the thin foils obtained from the as-received Ti-6Al-4V showed dislocation tangles and low density arrangements in the equiaxed alpha grains (Figure 14). The beta grains showed a "mish-mash" of unresolvable structure (Figure 15). The Ti-8Al-1Mo-1V thin foils showed the unaged alpha grain with isolated dislocations, aged alpha grains with a martensitic platelet structure, and beta particles with a striated appearance. Isolated particles of titanium hydride or oxide were evident (Figure 16). This would be normally expected in this kind of material.

The preferred orientation in each material was determined by X-ray diffraction techniques. The 2024-T3 aluminum had a primary orientation of the [200] direction and a secondary orientation of the [111] parallel to the rolling direction. The diffraction pattern from the Ti-8Al-1Mo-1V showed primarily the [1011] direction and, to a lesser degree, the [1010] direction of the HCP alpha phase in the rolling direction. The preferred orientation of the Ti-6Al-4V alloy was found to consist primarily of the basal plane direction [0002] of the HCP phase in the rolling direction.

From these examinations, it was concluded that the three materials for a program were representative of the condition and heat treatment for each alloy.

Thermal Exposure Specimens - Optical and electron metallography examinations of specimens of 2024-T3, Ti-6Al-4V, and Ti-8Al-1Mo-1V revealed no notable changes in microstructure. However, since large changes were produced in the tensile behavior of the 2024-T3 and Ti-8Al-1Mo-1V, transmission electron microscopy was used to examine changes in the precipitate structure.

Specimens of 2024-T3 exposed to 300F for 50, 250, and 500 hours were electrolytically thinned to prepare foils for TEM. Thermal exposure of 50 hours at 300F might be expected to show a resolvable aluminum-copper precipitate. The microstructure (Figure 17) revealed a phase identified by selected area diffraction as θ''. Both well-defined and ill-defined structures were seen in the thin foils. The microstructure of the 250-hour exposure showed transformation of some θ'' to θ' phase and, possibly, some growth of the large equilibrium θ particles ($CuAl_2$)(Figure 18). This also was determined with the use of selected area electron diffraction analysis. After 500 hours exposure, there were no changes in the phases present. However, the platelets of θ' grew thicker and were much more clearly resolved (Figure 19).

Examination of specimens of Ti-6Al-4V exposed to 800F for 50 hours and 500 hours revealed no notable changes in the substructure as compared to the as-received material.

The thermal exposure specimen of Ti-8Al-1Mo-1V held at 800F for 50 hours showed the same substructure as the as-received specimen (Figure 20). However, the microstructure of the specimen exposed 500 hours revealed a very fine spherical precipitate in the aged alpha grains (Figure 21). Selected area electron diffraction patterns from these particles indicated the presence of the precipitate identified as α_2-Ti_3Al. The particles are approximately 0.05 μ in diameter.

Creep Strained Specimens - Optical and electron metallographic examinations were performed on the 50-hour and 500-hour specimens strained to 1% creep. Changes in the size and amount of precipitate in the 2024-T3 were observable. However, the Ti-6Al-4V and Ti-8Al-1Mo-1V did not show notable changes.

Thin foils of 2024-T3 from specimens creep strained to 1% strain at 300F at 50 hours and 500 hours were examined by transmission electron microscopy (TEM). The formation of θ'' (as identified by selected area electron diffraction) was evident. This is seen in Figures 22 and 23. In comparison to the precipitate structures from the 50-hour thermally exposed specimen, the microstructure of the strained specimen consists of both θ'' and θ', indicating a more advanced aging. Resolvable platelets of θ' or θ' plus residual θ'' were present. A definite growth of the incoherent θ phase was also evident.

TEM examination of the specimens of Ti-6Al-4V strained to 1% creep in 50 and 500 hours showed no significant sensitivity to the strain rate. The strain accumulation evidenced itself in occasional areas of slip and cross slip reactions plus a small amount of twinning, as seen in Figures 24 and 25.

TEM examination of the Ti-8Al-1Mo-1V specimen strained to 1% creep in 50 hours showed isolated areas of subcell formation which were not seen in the as-received or thermal exposure specimens (Figure 26). In general, however, only subtle differences in microstructure were noted for this material as a result of the 1% deformation process. A Ti-8Al-1Mo-1V specimen exposed at 800F for 500 hours contained Ti_3Al as shown by TEM and electron diffraction. A somewhat greater amount of Ti_3Al was found in the specimen strained to 1% in 500 hours. The Ti_3Al was formed in the aged alpha grains near the intersection of the Martensite striation and the grain boundary.

Scanning electron fractography was performed on the fracture faces of the two tensile coupons of Ti-8Al-1Mo-1V. One specimen was thermally exposed 500 hours at 800F, and the other was creep strained to 1% strain in 623 hours. Both specimens were tested at room temperature. A change in the fracture topology was noted in that the thermally exposed specimens (Figure 27) showed primarily a dimpled rupture and the strained specimen a brittle type quasi-cleavage fracture.

DISCUSSION

The 2024-T3 aluminum-base alloy was chosen specifically for this program because of its instability at elevated temperatures. The T3 condition consists of solution heat treating, cold working through a straightening process, and then naturally aging to a substantially stable condition. The T4 condition is the same, except for omission of the cold straightening operation. The T81 and T6 conditions are like T3 and T4, with the aging done instead at 375F, which results in a more thermally stable precipitate structure.

In the solution-treated and room-temperature-aged condition, the 2024 alloy presents a microstructure of G.P. zones, usually spherical in nature[3,4]. Upon aging at elevated temperatures, the Θ" ($CuAl_2Mg$) precipitates form solid solution in a Widmanstätten pattern. This coherent phase continues growth to the Θ' phase. It has also been reported[5] that in the 2024 alloy only a single phase results called S'. However, diffraction results from this study indicate a Θ" intermediate step. Further aging results in formation of the incoherent Θ phase.

The alloy in the T-3 condition shows a substructure of small G.P. zones that appear spherical in electron micrographs (Figure 13). G.P. zones, which result from room temperature aging, are formed by copper and magnesium atoms clustering apparently on the (110) planes of the aluminum and are approximately 500Å in size. After 50 hours of thermal exposure at 300F, the microstructure appears to be that of the ordered phase Θ" as reported by Thomas and Washburn[4]. The appearance was of an ill-defined structure; both the G.P. zones and Θ" exist simultaneously, but the Θ" phase is more resolvable.

It has been reported that as the amount of Θ" phase reaches its maximum, maximum strength is also achieved. This would closely correlate with the tensile property behavior.

Examination of the mechanical properties of the thermal exposure specimens revealed that such aging process (or processes) had occurred (Table III, Figure 3). After 50 hours exposure, both the yield strength and the ultimate tensile strength increased appreciably at both ambient temperature and 300F. The elongation decreased in this period of time from 19% to 12%. The microstructure also reveals the precipitation of Θ" and the lessening of the number of G.P. zones (Figure 17). After 250 hours of exposure, the yield strength increased appreciably at both room temperature and 300F. The ultimate tensile strength at 250 hours increased slightly at room temperature and decreased slightly at 300F. The ultimate tensile strength of the 500-hour specimens were essentially the same as those of the 250-hour specimens at room temperature and 300F. The elongation for 250 and 500 hours specimens continued to decrease to 8% and 5% at 500 hours. The decrease in elongation may indicate a formation of greater amounts of incoherent precipitate. The microstructure of the 250-hour exposure did show an increased growth in the platelet structure of the coherent Θ' phase plus equilibrium Θ.($CuAl_2Mg$) phase (Figure 15). After 500 hours exposure,

there were no further changes in the precipitate morphology; however, the platelets of Θ' grew thicker and were much more clearly resolved (Figure 19). It did appear that there was also a greater amount of incoherent Θ phase present, which may explain the decrease of ductility.

Creep straining to 1% strain in 50 and 250 hours at 300F had essentially the same effect on tensile properties as thermal exposure for the same time (Table III, Figure 6, 7); that is, a considerable increase in yield strength, a small increase in tensile strength, and a large decrease in elongation.

Examination of the precipitate character of specimens strained to 1% strain in 50 hours revealed a structure similar to the thermal exposure condition. However, the extent of the aging process appeared to be more advanced in that the Widmanstätten needles are more well defined, longer, and broader. Selected area electron diffraction did not indicate the presence of G.P. zones. After straining to 1% in 250 hours, there is a growth in the Θ' $CuAl_2Mg$ phase and more of the Θ phase, indicating a slightly overaged condition.

Tensile tests of specimens strained to 1% in a nominal 500 hours, however, showed a large decrease in both ultimate and yield strengths. The ultimate and yield tensile strengths are nearly equal after creep straining. The elongation is about the same as after a 250-hour prestraining. This probably signifies that both thermal exposure and creep straining are contributing to the change in tensile properties. The radical drop in ultimate and yield tensile strengths after 400 hours would signify an overaging situation where the precipitation strengthening phases Θ" and Θ' are now decreasing and the increase in incoherent phase Θ could now be providing large crack initiation sites. The specimen prestrained 1% in 400 hours showed a considerably overaged structure. The platelets appeared to be elliptical in nature rather than needle-like. (Figure 23)

The amount of the incoherent phase Θ is definitely more substantial. Random areas of dislocation pinning were evident in the as-received and 50-hour specimens, but in specimens strained for longer times, the effects were diminished.

These results agree with similar work performed by Gluck, Voorhees, and Freeman(6) on the effects of creep strain accumulation on the tensile behavior of 2024-T86. In that program, also, significant changes occurred as a result of thermal exposure, but increased changes in tensile behavior were brought about by creep strain accumulation. It is interesting to note that the relatively stable 2024-T86 shows similar effects to the behavior in the much less stable T3 condition.

Of the three alloys studied in this program, the Ti-6Al-4V material is reported to be the most stable. The Ti-6Al-4V alloy is an alpha-beta type alloy. In the solution-treated condition, the microstructure consists of equiaxed alpha grain structure with small globular or acicular

beta particles at the grain boundaries of the equiaxed alpha grains. Examination of the thin foils of as-received Ti-6Al-4V revealed nothing unusual. The beta particles showed the effects of original processing; that is, isolated dislocation pileups and slip bands. The alpha grains showed the transformation from beta to alpha (martensitic structure). No evidence of impurities was detected in the microstructure of several foils examined. The tensile results from specimens exposed to 50, 250, and 500 hours at 800F show insignificant changes in the yield, ultimate, and elongation properties of the Ti-6Al-4V alloy (Table IV, Figure 4).

Specimens exposed for 50 hours at 800F showed a microstructure identical with that of the as-received condition. The 500-hour specimen also revealed a microstructure similar to the as-received condition. Apparently, no aging processes or stress relievement in terms of dislocation changes are occurring at this temperature. After creep straining to 1% strain in 50, 250, and 500 hours, essentially no change in the yield or ultimate tensile strength was observed (Table IV, Figure 8, 9). A very slight increase in the ultimate tensile strength appeared to be taking place. A small decrease of 2% in elongation appeared after 250 hours and, subsequently, in the 500-hour thermal exposure.

The high magnification microstructure of the specimen of Ti-6Al-4V strained to 1% creep in 50 hours showed the strain accumulation evidencing itself in occasional areas of slip and cross slip. There was also a definite indication of an increase in twinning occurring. The microstructures, after 250 hours and 500 hours to 1% strain, showed the same deformation reactions that had occurred in the 50-hour specimen, that is, slip, cross slip, and twinning. The alloy appears to have remained stable through both thermal exposure and creep straining. The presence of slip and twinning would be expected to a small degree due to the amount of deformation imposed upon the specimen.

The Ti-8Al-1Mo-1V alloy is a super-alpha type titanium-based alloy. In the duplex-annealed condition (8 hours at 1450F, furnace-cooled, plus 15 minutes at 1450F, air cooled), the structure consists of two phases, alpha and beta. The alpha phase consists of two types of grains, equiaxed and striated-aged grains. Much less beta phase is present than in the Ti-6Al-4V alloy. The high aluminum content produces the primary strengthening in the alpha phase.

Thermal instability has been reported to exist in the Ti-8Al-1Mo-1V alloy[7,8,9,10,11]. Long-time exposures as low as 600F have resulted in a lowering of the ductility and a subsequent increase in yield strength. This has been reported to be associated with an order/disorder reaction in the alloy or the precipitation of a super-lattice type phase designed at α_2-Ti_3Al. Examination of the structure of the as-received Ti-8Al-1Mo-1V revealed unaged alpha grains with isolated dislocations, aged alpha grains with a martensitic platelet structure, and beta particles with a striated appearance. Isolated particles of titanium hydride or oxide were evident. This would be normally expected

in this grade of material. The tensile properties obtained from the thermal exposure specimens revealed very slight changes in the yield and ultimate tensile strengths for specimens exposed for up to 500 hours. There did appear to be a small decrease in ductility after 500 hours exposure in 800F and a slight increase in yield strength.

A specimen exposed to 800F for 50 hours showed the same structure as the as-received material. However, the microstructure of the Ti-8Al-1Mo-1V, exposed 500 hours at 800F, revealed a very fine spherical precipitate occurring in the aged alpha grains. Selected area electron diffraction patterns in these particles indicated the precipitate to be $_2$-Ti_3Al. Particles were approximately 0.05 microns in diameter. The formation of the $_2$-Ti_3Al phase is usually associated with a loss of ductility and an increase in yield tensile strength.

The specimens prestrained to 1% strain in creep at 800F showed slight increases in yield and ultimate strength with essentially no change in elongation at 250 hours. Examination of the thin foils from specimens strained to 1% strain in 50 hours did not reveal a substructure different from the thermal exposure specimen. After 250 hours to obtain 1% strain, the presence of the ordered $_2$-Ti_3Al appeared. Specimens prestrained to 1% in 500 hours had essentially the same yield and ultimate strengths as specimens exposed for 500 hours at 800F but not strained. However, the 500-hour prestrained specimens showed significant decreases in the elongation. The ambient temperature tested specimens showed a drop from 19% to 13%, and the 300F elongation decreased from 16.5% to 13.5%. After 500 hours, a significant amount of the ordered phase was present. This would reasonably explain the loss in ductility.

Changes in residual strength observed in the 2024-T3, Ti-6Al-4V, and Ti-8Al-1Mo-1V represent a function of creep rate over the range from 2×10^{-4} in/in per hour to 2×10^{-5} in/in per hour. From these data, there does not appear to be a single relationship between strain rate and residual strength. The changes in tensile properties appear to be explainable in terms of microstructural mechanisms. The results from these studies positively indicate that the hypothesis that "the accumulation of a constant strain under various temperatures and various stresses results in equivalent damage" is not universally valid. Materials strained to a 1% strain level at two different strain rates may have different tensile properties. If the hypothesis were true, no change could be anticipated. The only alloy which appears to support this hypothesis is the Ti-6Al-4V alloy. However, tensile properties are a low sensitivity method of measuring damage. Fatigue life or other mechanical properties might reveal failure of the hypothesis for Ti-6Al-4V as well.

CONCLUSIONS

1. The use of creep-strain accumulation as a criterion for obtaining equivalent damage during an accelerated elevated temperature creep test is not universally valid.

2. The tensile properties of 2024-T3, Ti-6Al-4V, and Ti-8Al-1Mo-1V are affected by both thermal exposure and creep straining at elevated temperatures.

3. The tensile property behavior of 2024-T3 after thermal exposure and also after creep straining is explainable in terms of microstructural aging processes.

4. The loss of ductility in Ti-8Al-1Mo-1V after long-time exposures at 800F is related to the presence of the α_2-Ti_3Al phase.

5. The Ti-6Al-4V alloy is the most stable alloy during either thermal exposure or creep straining.

6. Microstructure can successfully be used to correlate residual strength with thermal exposure changes and creep straining damage in these alloys.

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9. "Instability and Evidence of Ordering in Ti-8Al-1Mo-1V Alloy," P. J. Soltis, Unpublished Paper, Naval Air Engineering Center.

10. "Crystal Structure of Ti-Al," Pol Duwez and Jack L. Taylor, Journal of Metals, Vol. 4, page 70, January 1952.

11. "Examen per Micrographs at Microdiffraction Electroniques Sur Coupes Minces d'Alliages de Titanea 7, 10 et 15% d'Aluminum," Memoires Scientifiques de la Rev. Metallurg. LVI, No. 4, Page 118, 1959.

TABLE I

CHEMICAL ANALYSIS OF AS-RECEIVED MATERIALS

2024-T3

Cu, %	Si, %	Fe, %	Mn, %	Mg, %	Cr, %
4.41	0.39	0.35	0.61	1.51	0.07

Ti-8Al-1Mo-1V, Duplex Annealed

Al, %	Mo, %	V,%	C, %	N, %
8.2	1.1	0.98	0.05	0.02

Ti-6Al-4V Annealed

Al, %	V, %	C, %	N, %	Fe, %
6.4	4.1	0.06	<0.05	0.19

TABLE II

TENSILE PROPERTIES OF AS-RECEIVED MATERIALS

SAMPLE	TEMP.	YTS (0.2% PSI)	UTS (PSI)	ELONGATION (%)
Mechanical Properties of Ti-6Al-4V, Annealed				
6-1	RT	137,000	144,300	17.0
6-2	RT	139,000	145,400	17.0
6-3	RT	135,400	142,800	15.0
6-4	800F	81,500	97,400	15.5
6-5	800F	80,200	96,800	16.0
6-6	800F	79,900	97,400	16.5
Mechanical Properties of Ti-8Al-1Mo-1V, Duplex Annealed				
8-1	RT	141,000	151,900	20.5
8-2	RT	138,600	149,000	19.0
8-3	RT	138,900	148,700	19.0
8-4	800F	86,700	106,900	17.5
8-5	800F	86,400	106,800	16.5
8-6	800F	84,500	104,800	15.5
Mechanical Properties of 2024-T3				
1A-103	RT	53,600	70,800	21.0
1A-107	RT	53,200	70,900	22.0
1A-108	RT	54,200	71,000	22.0
1A-109	300F	49,500	61,600	25.5
1A-124	300F	50,800	61,800	28.0
1A-133	300F	49,300	61,800	26.0

TABLE III

COMPARISON OF TENSILE PROPERTIES OF 1% CREEP STRAINED SAMPLES OF 2024-T3

SAMPLE NO.	CREEP LOAD (KSI)	STRAIN (%)	CREEP EXPOSURE AT 300°F, HOURS	TEST-TEMP (°F)	YTS (PSI)	UTS (PSI)	ELONGATION (%)
1A208	51	1.01	23	RT	58,000	74,000	14.0
1A209	51	1.02	48	RT	57,330	74,250	17.0
1A209	51	1.0	78	RT	64,240	75,600	13.0
1A216	51	0.94	41	300	62,710	67,050	18.0
1A210	51	1.10	48	300	54,400	64,940	16.0
1A214	51	0.91	49	300	56,280	65,490	16.0
1A203	47	1.23	305.2	RT	68,700	73,900	10.0
1A215	47.5	1.0	310.4	RT	70,200	74,900	12.1
1A218	47.5	0.72	425	RT	70,300	75,100	12.1
1A224	48	0.90	250	300	64,000	66,700	7.0
1A220	47.5	1.13	329	300	59,900	60,000	5.5
1A223	48	0.78	291	300	62,300	63,900	10.0
1A205	46	1.31	406	RT	69,900	71,000	4.0
1A221	47	0.61	498	RT	70,800	72,000	6.0
1A211	46	1.06	408	RT	70,100	71,200	7.0
1A227	47.5	0.95	420	300	52,400	53,500	9.0
1A222	47.5	1.30	402	300	52,200	53,200	9.0
1A230	47	0.98	406	300	49,400	51,700	12.5
1A216	51	0.95	41.2	Micro Specimen			
1A226	48	0.61	297	Micro Specimen			
1A219	47.5	0.86	604	Micro Specimen			

TABLE IV

TENSILE PROPERTIES OF 1% CREEP STRAINED SAMPLES OF Ti-6Al-4V

SAMPLE NO.	CREEP LOAD (KSI)	STRAIN (%)	CREEP EXPOSURE AT 800°F, HOURS	TEST-TEMP (°F)	YTS (PSI)	UTS (PSI)	ELONGATION (%)
6-10	67	0.97	19.8	RT	132,600	144,100	13.0
6-11	66	1.05	21.1	RT	133,200	144,400	13.0
6-6	68	1.2	40	RT	134,300	144,300	14.0
6-4	70	1.9	88	800	82,800	98,700	16.0
6-8	68	1.8	23.6	800	80,400	97,100	15.0
6-13	65	1.2	19.0	800	80,900	98,600	16.0
6-16	54	0.78	265.7	RT	131,900	143,900	13.0
6-20	55	0.97	194.7	RT	131,600	144,200	14.0
211-2	60	0.70	225	RT	131,300	142,900	13.0
6-20	55	0.81	195	800	76,400	97,000	15.0
6-22	55	0.95	247	800	78,800	98,100	15.0
212-2	55	0.91	196	800	78,700	98,100	17.0
221-1	54	0,86	437	RT	132,100	143,100	13.0
6-15	55	0.99	491	RT	133,400	144,400	14.0
221-2	55	0.95	487	RT	132,900	141,900	13.0
6-12	54	0.95	500	800	76,800	96,900	15.0
6-19	55	0.82	409	800	79,200	98,500	16.0
6-17	55	0.90	487	800	82,700	100,800	14.5
6-14	67	0.90	49	Micro Specimen			
6-18	55	1.02	180.2	Micro Specimen			
211-3	55	0.7	400	Micro Specimen			

TABLE V

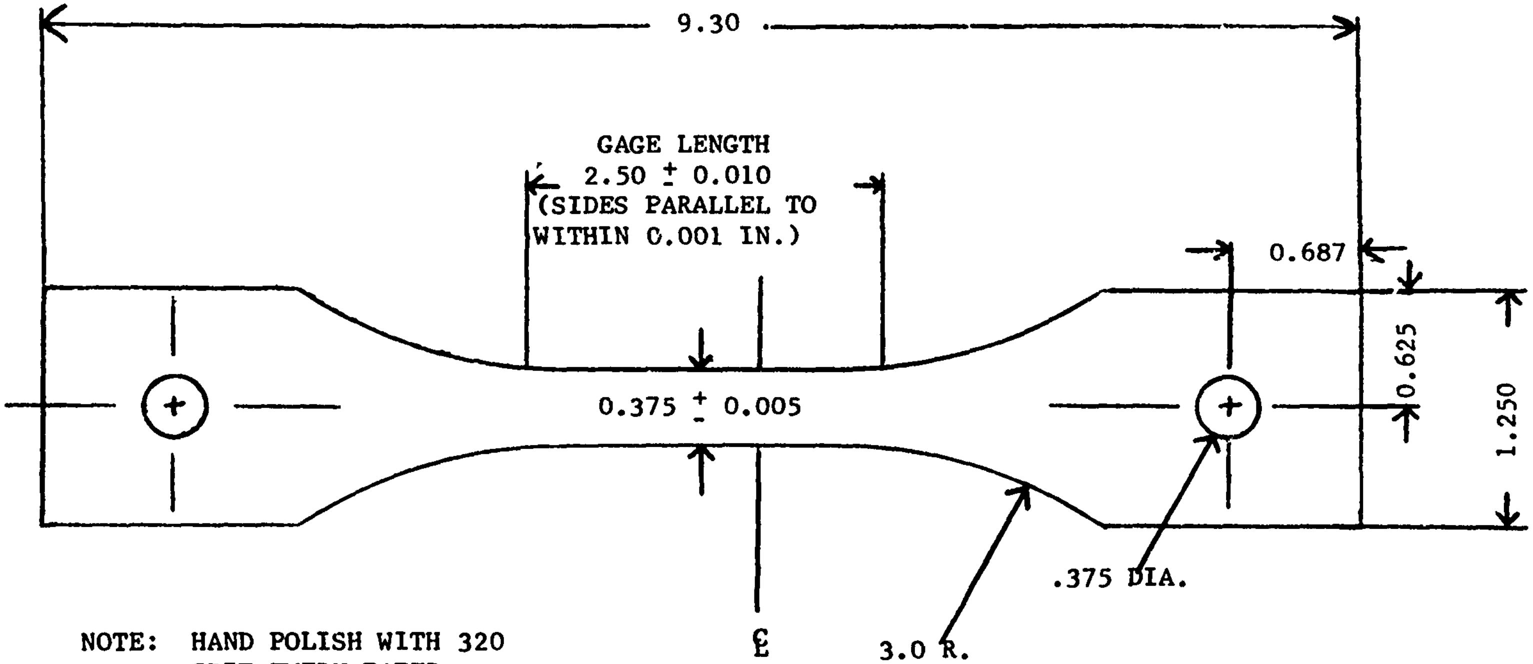
TENSILE PROPERTIES OF 1% CREEP STRAINED SAMPLES OF Ti-8Al-1Mo-1V

SAMPLE NO.	CREEP LOAD (KSI)	STRAIN (%)	CREEP EXPOSURE AT 800°F, HOURS	TEST-TEMP (°F)	YTS (PSI)	UTS (PSI)	ELONGATION (%)
8-7	73	0.79	49	RT	143,900	156,500	20.0
8-5	76	1.80	49	RT	142,900	157,000	17.0
8-6	73	1.20	56	RT	141,900	157,300	16.0
8-1	72	1.3	171	800	87,100	111,000	15.2
8-3	74	0.9	88	800	87,600	110,000	14.8
8-9	73	0.91	89	800	90,400	114,000	14.6
8-2	69	0.8	287	RT	144,800	158,000	20.0
8-11	70	0.84	247	RT	143,600	156,400	19.0
8-22	73	0.95	420	RT	141,900	153,600	20.0
8-18	73	0.72	216	800	89,100	113,200	15.0
8-20	73	1.11	289	800	88,800	111,400	18.0
8-21	73	0.91	406	800	90,800	114,100	16.0
8-8	69	0.83	522	RT	143,900	152,700	9.0
8-26	74	1.10	623	RT	144,900	155,000	12.0
8-15	74	0.81	629.5	RT	143,300	155,200	14.0
8-17	73	0.83	645.5	800	90,500	114,300	14.0
8-19	71	0.97	498.6	800	90,600	115,100	13.5
8-23	71	0.81	510	800	90,800	112,500	14.0
8-10	74	0.89	53	Micro Specimen			
8-14	71.5	1.02	340	Micro Specimen			
8-16	72	0.86	440	Micro Specimen			

TABLE VI

TENSILE PROPERTIES OF THERMALLY EXPOSED SAMPLES

MATERIAL	EXPOSURE	TEST-TEMP (°F)	YTS (PSI)	UTS (PSI)	ELONGATION (%)
2024-T3	As-received	RT	53,670	70,900	21.8
	50 hours 300F	RT	58,500	74,400	18.5
	250 hours 300F	RT	72,100	76,000	8.5
	500 hours 300F	RT	70,450	74,750	10.0
2024-T3	As-received	300	47,870	61,700	26.5
	50 hours 300F	300	55,800	65,000	19.5
	250 hours 300F	300	62,200	63,700	15.0
	500 hours 300F	300	62,100	65,500	14.5
Ti-6Al-4V	As-received	RT	137,130	144,170	16.3
	50 hours 800F	RT	134,500	142,500	17.0
	250 hours 800F	RT	135,500	142,500	16.5
	500 hours 800F	RT	135,400	142,500	16.5
Ti-6Al-4V	As-received	800	80,530	97,200	16.0
	50 hours 800F	800	79,450	96,350	18.0
	250 hours 800F	800	80,000	95,500	18.0
	500 hours 800F	800	79,300	96,400	16.5
Ti-8Al-1Mo-1V	As-received	RT	139,500	149,900	19.5
	50 hours 800F	RT	140,500	152,700	19.0
	250 hours 800F	RT	140,500	153,000	20.5
	500 hours 800F	RT	144,100	156,500	20.0
Ti-8Al-1Mo-1V	As-received	800	85,870	106,160	16.5
	50 hours 800F	800	88,250	109,800	15.0
	250 hours 800F	800	89,700	112,700	15.5
	500 hours 800F	800	88,100	111,500	16.5



NOTE: HAND POLISH WITH 320 GRIT EMERY PAPER

(Note: All dimensions in inches)

FIGURE 1 SPECIMEN CONFIGURATION FOR CREEP AND TENSILE TESTS

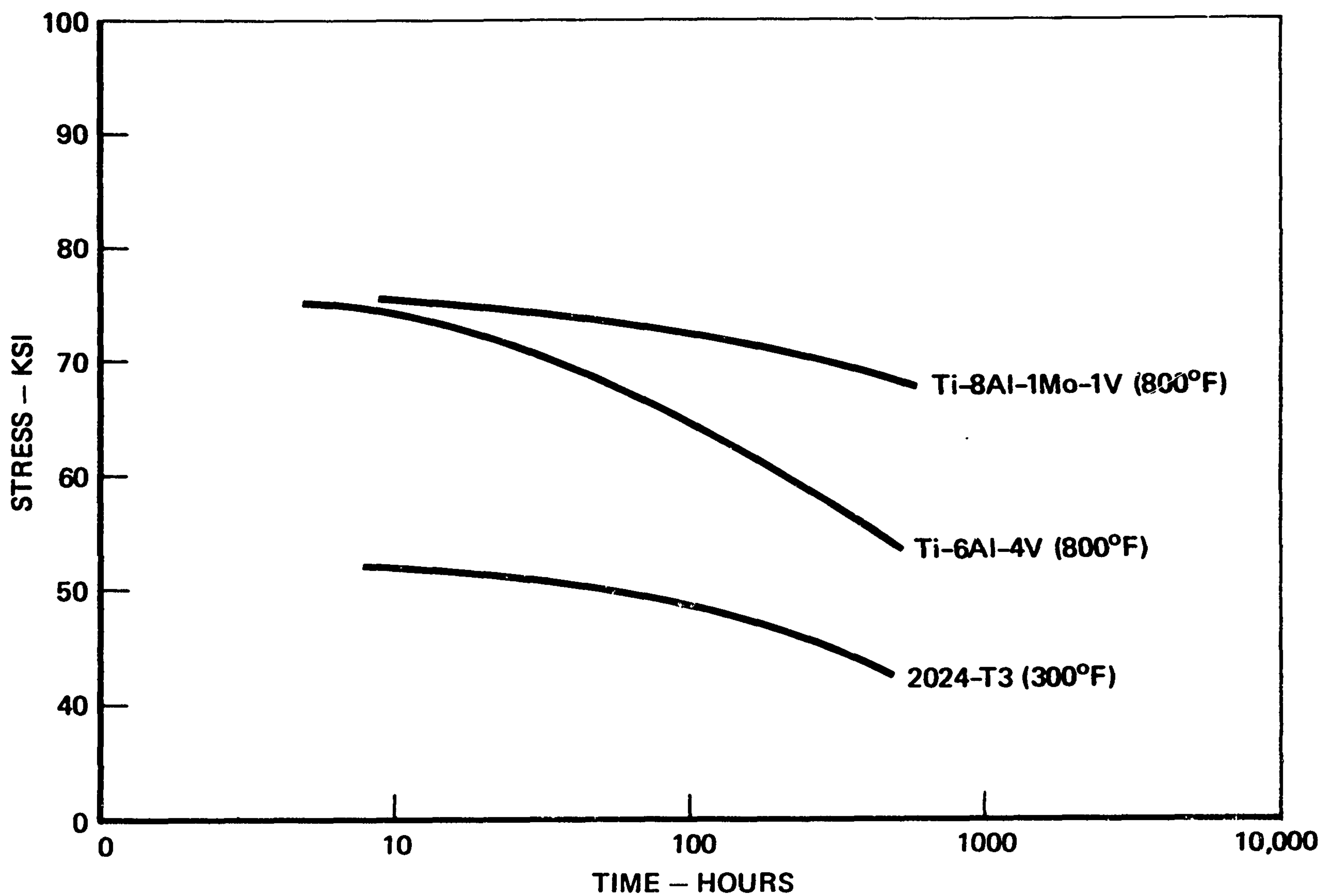


FIGURE 2. STRESS VS TIME TO OBTAIN 1% PLASTIC STRAIN IN CREEP

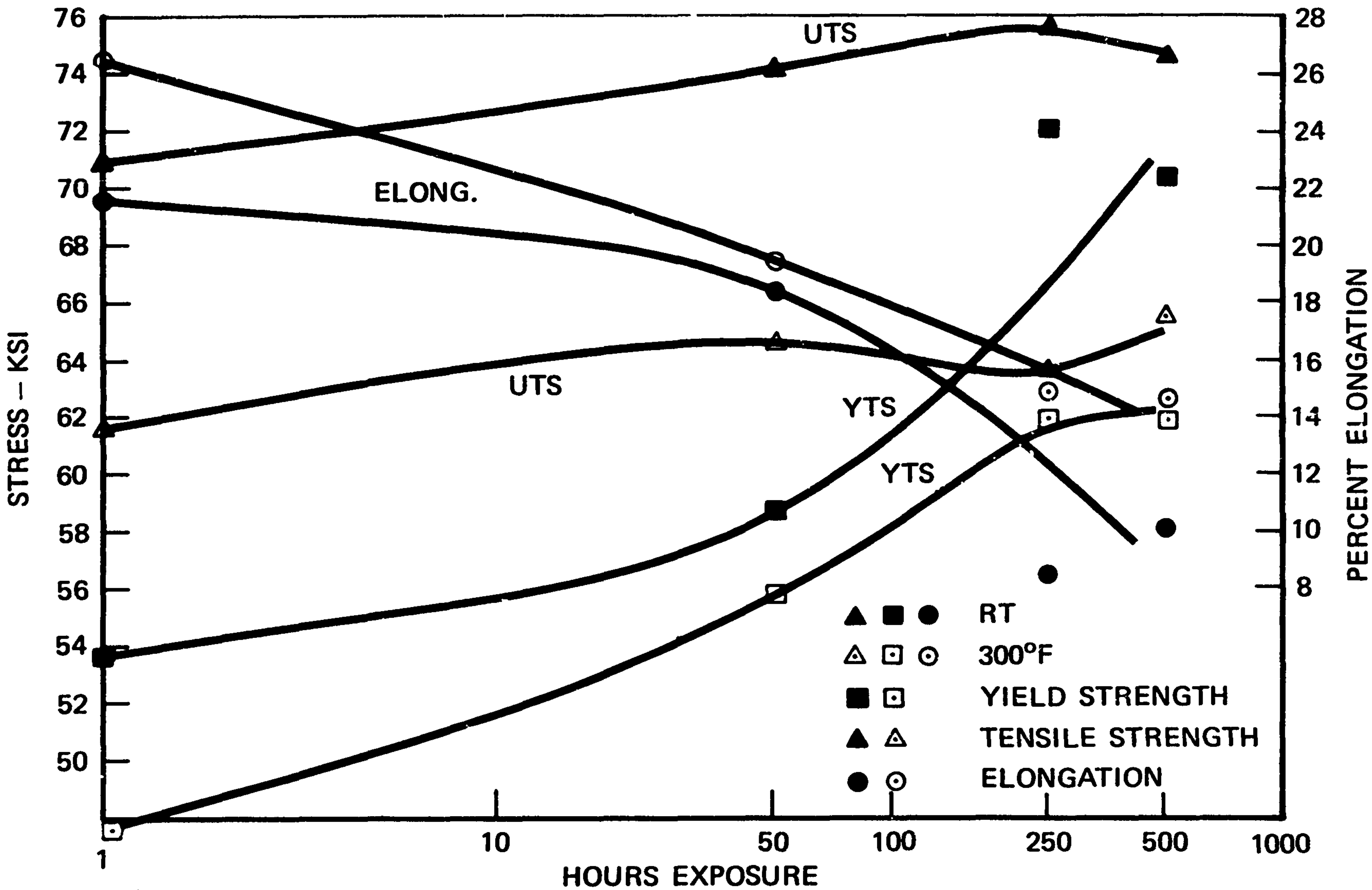


FIGURE 3. TENSILE PROPERTIES OF 2024-T3 AFTER THERMAL EXPOSURE AT 300°F

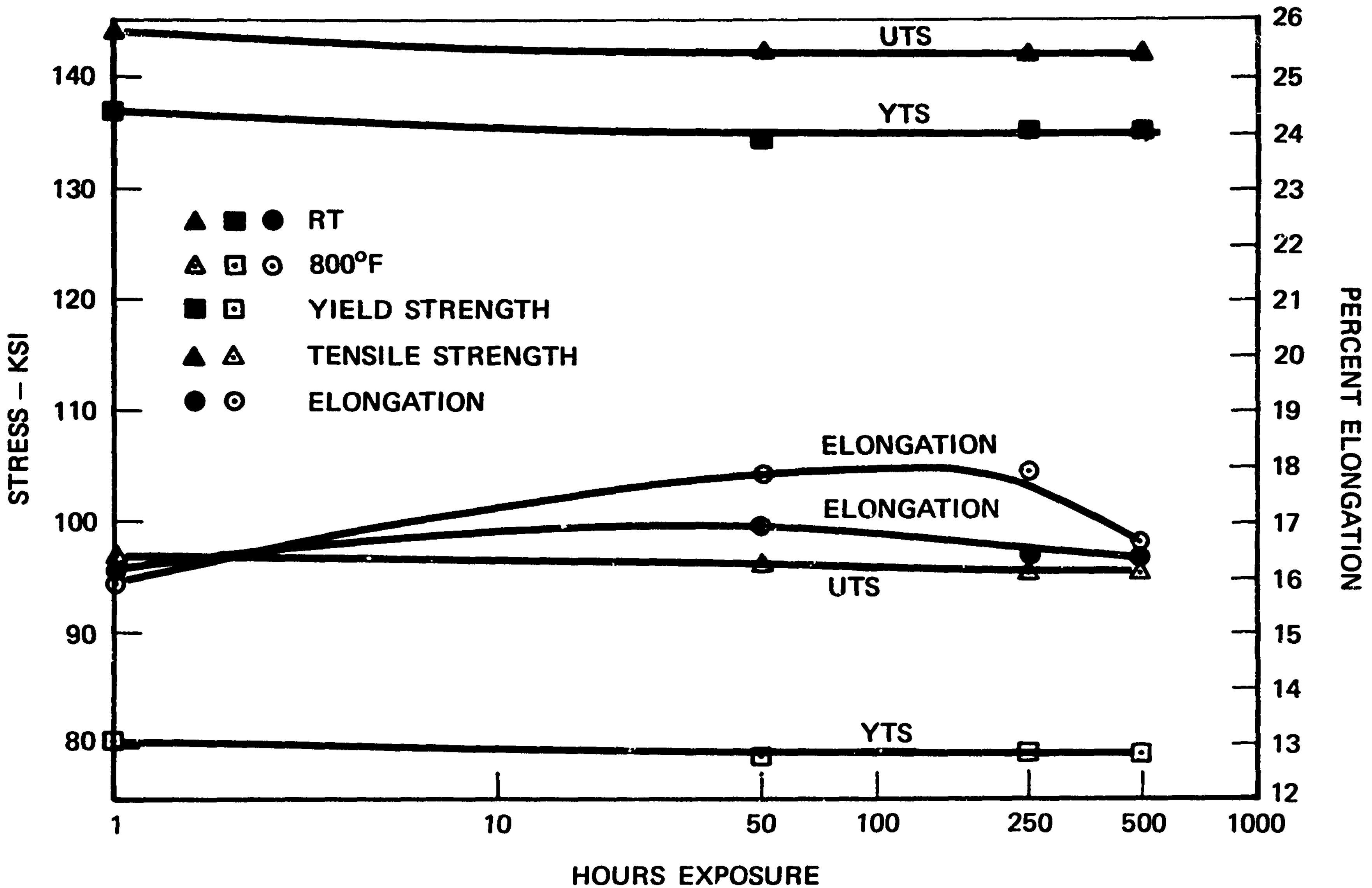


FIGURE 4. TENSILE PROPERTIES OF Ti-6Al-4V, ANNEALED AFTER THERMAL EXPOSURE AT 800°F

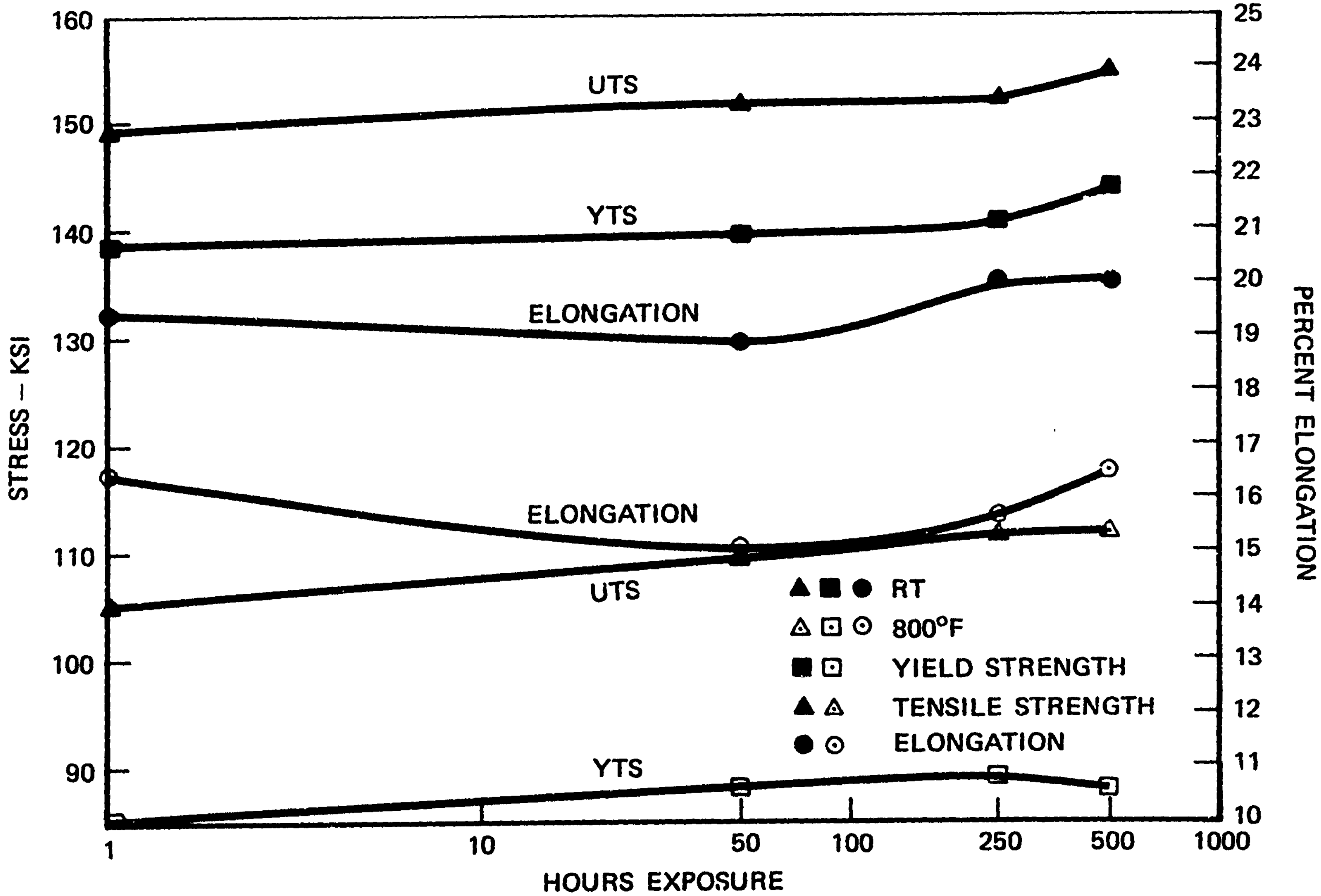


FIGURE 5. TENSILE PROPERTIES OF Ti-8Al-Mo-1V, DUPLEX ANNEALED, AFTER THERMAL EXPOSURE AT 800°F

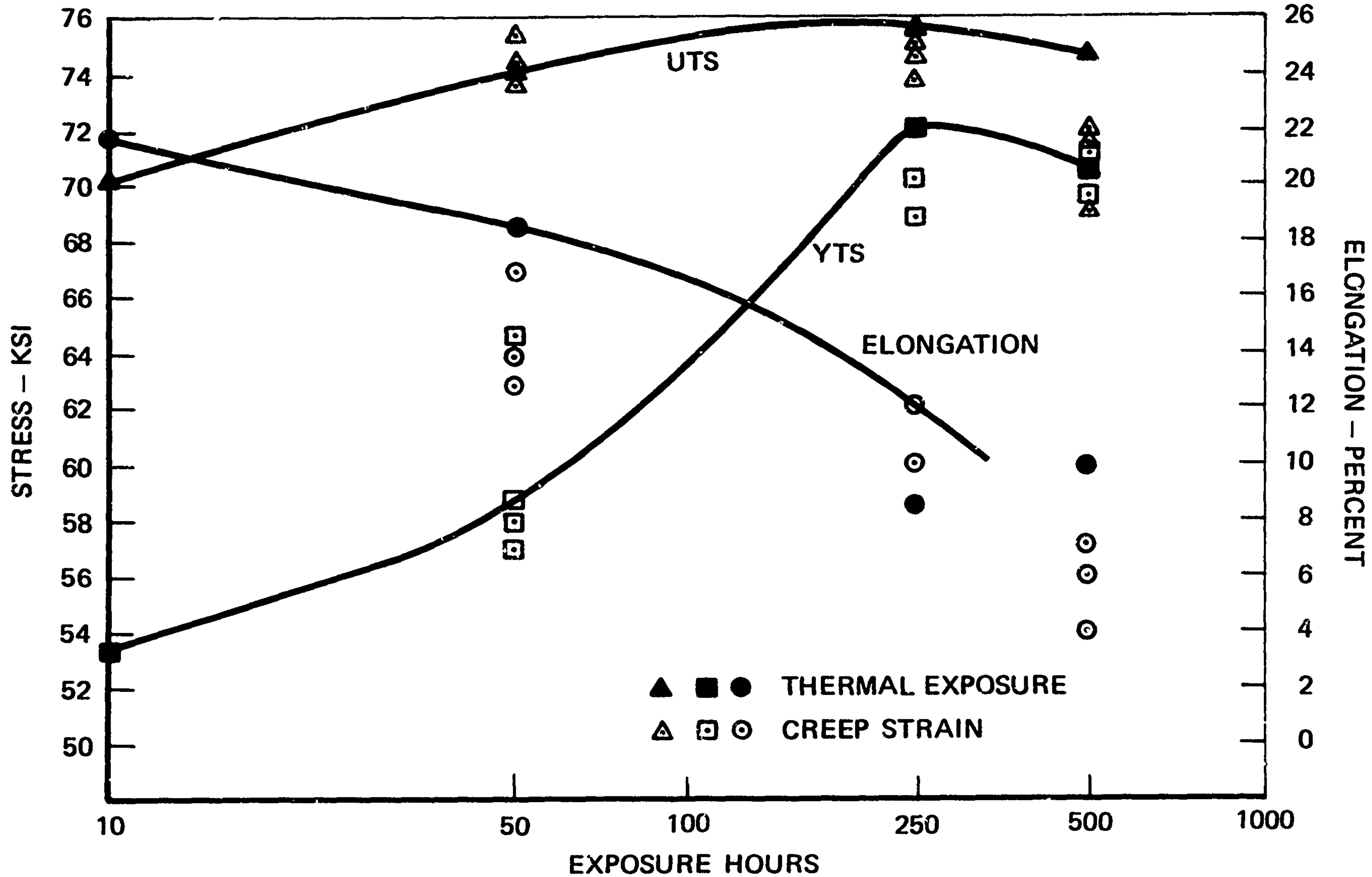


FIGURE 6. ROOM TEMPERATURE TENSILE PROPERTIES OF THERMALLY EXPOSED AND OF CREEP STRAINED 2024-T3

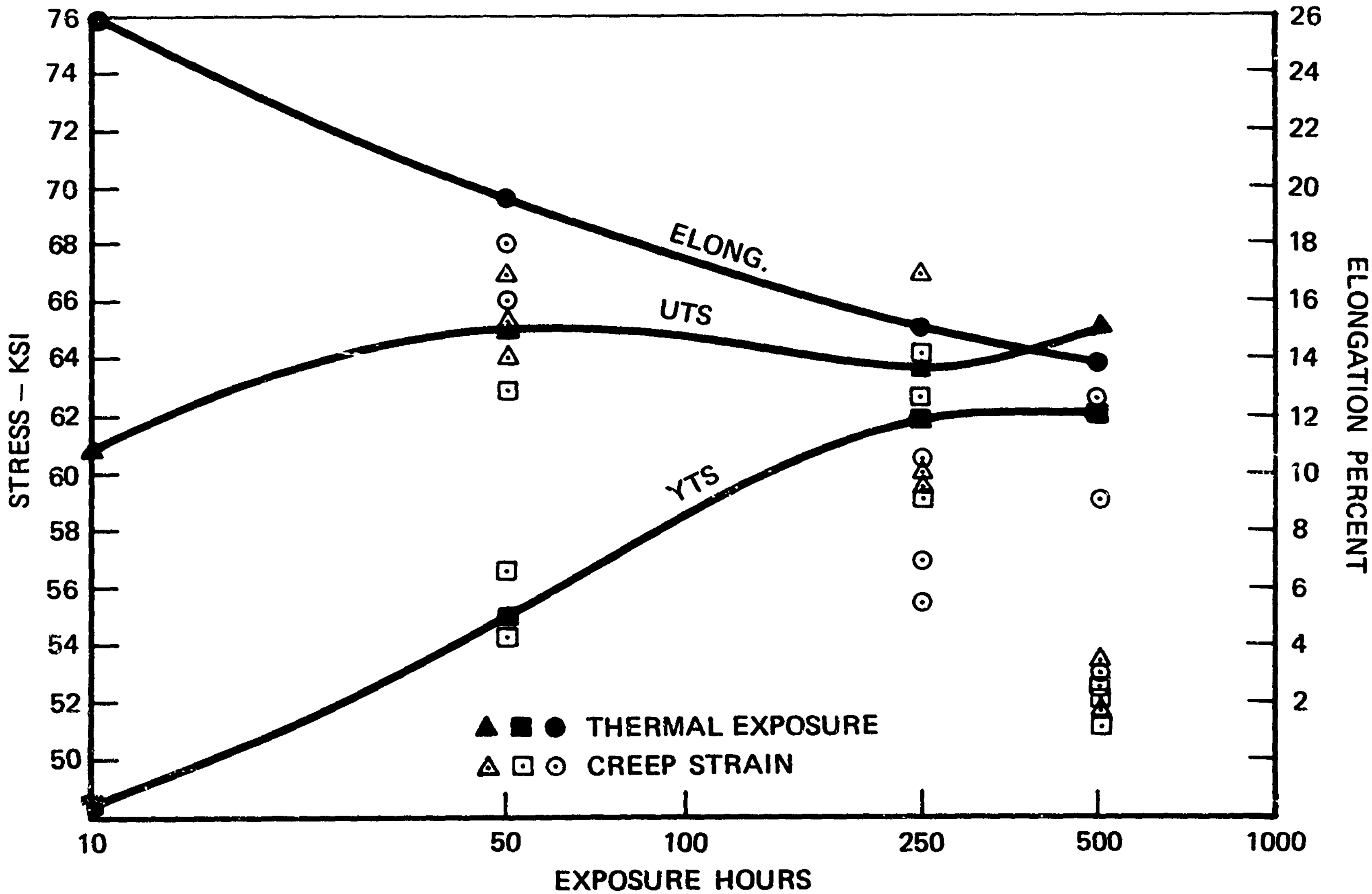


FIGURE 7. TENSILE PROPERTIES OF THERMALLY EXPOSED AND CREEP STRAINED 2024-T3 AT 300°F

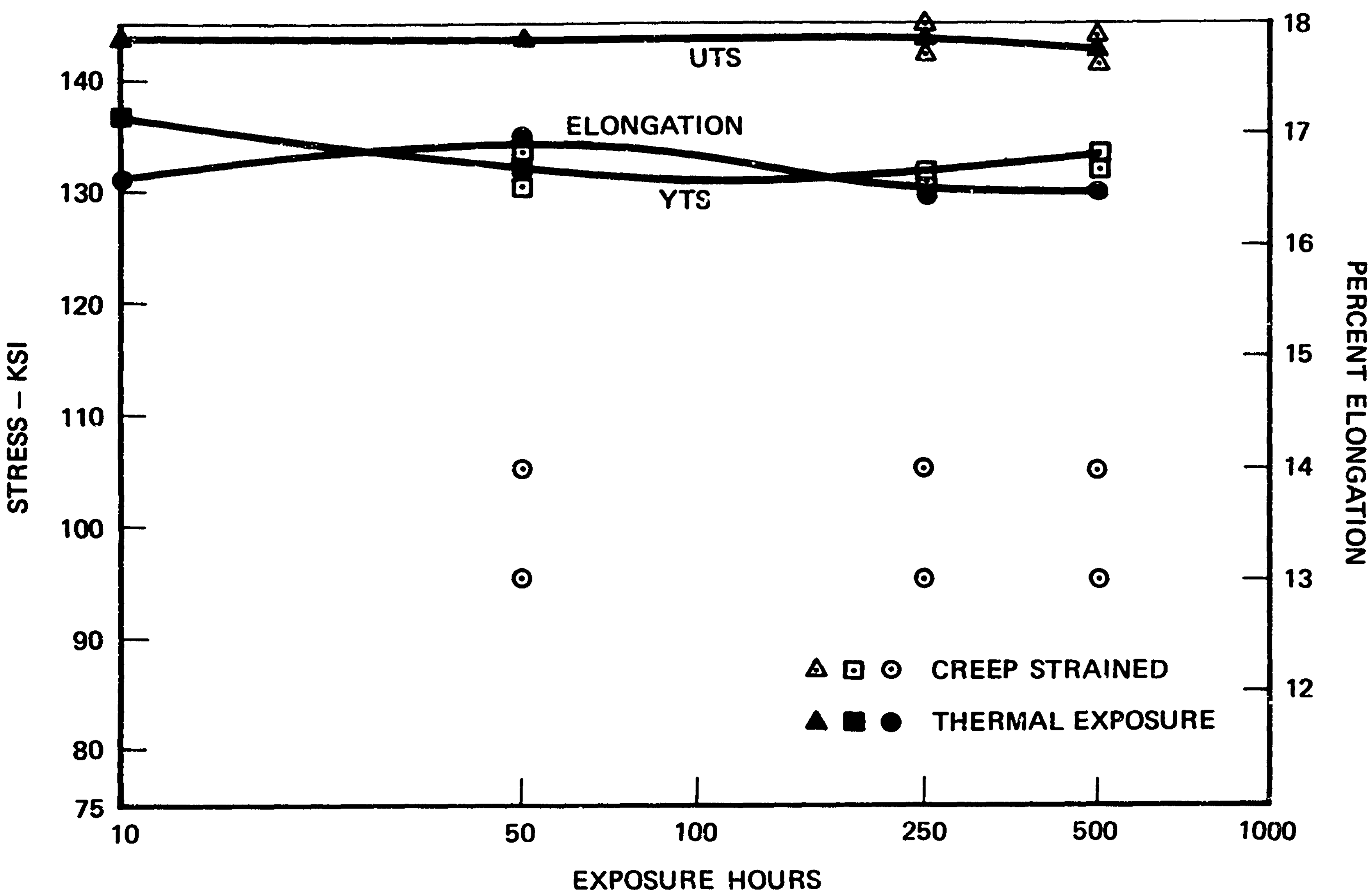


FIGURE 8. COMPARISON OF (ROOM TEMPERATURE) TENSILE PROPERTIES OF THERMAL EXPOSURE AND CREEP STRAINING OF Ti-6Al-4V

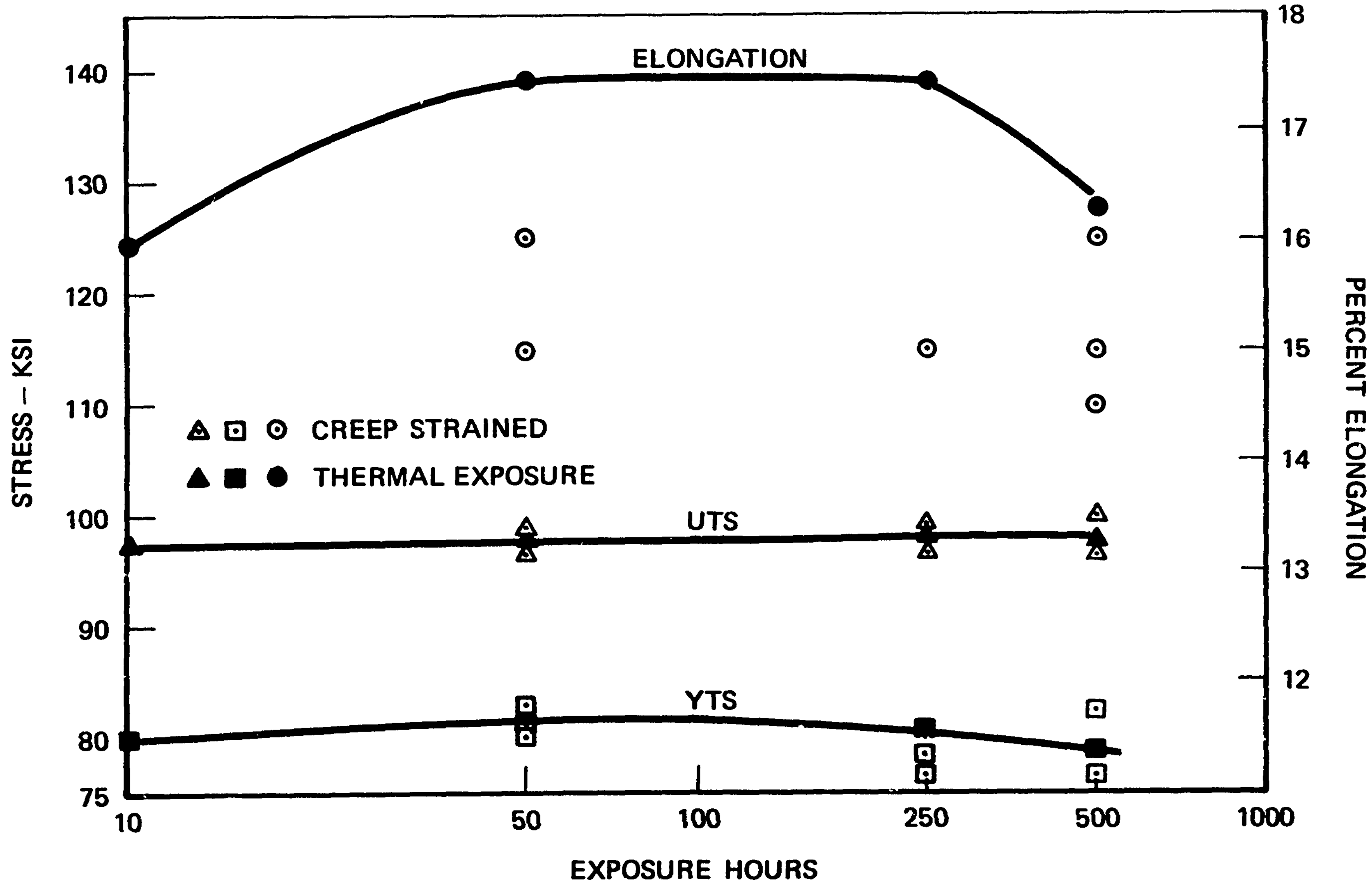


FIGURE 9. COMPARISON OF (800°F) TENSILE PROPERTIES OF THERMAL EXPOSURE AND CREEP STRAINING OF Ti-6Al-4V

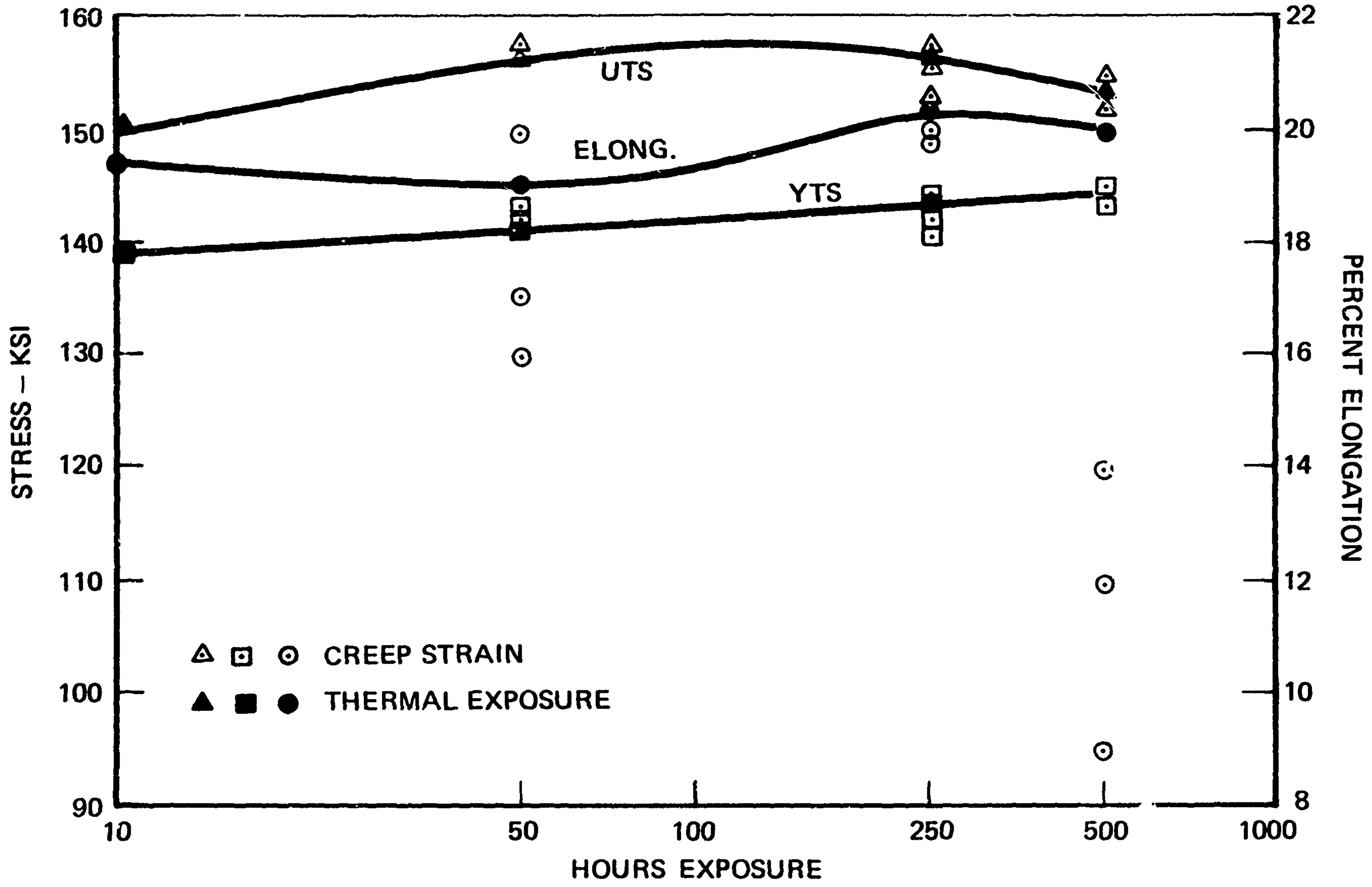


FIGURE 10. ROOM TEMPERATURE TENSILE PROPERTIES OF THERMALLY EXPOSED AND CREEP STRAINED Ti-8Al-1Mo-1V

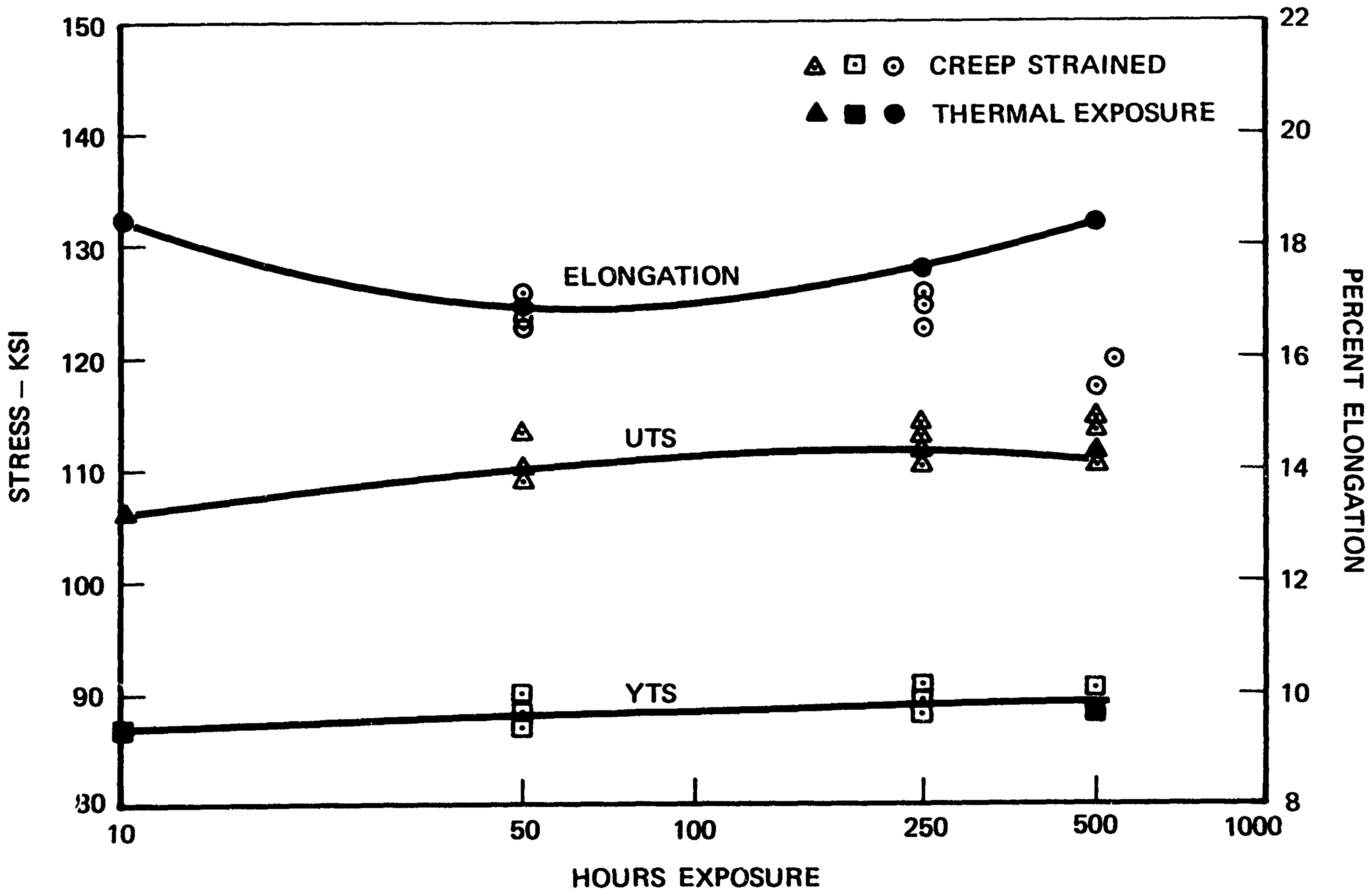
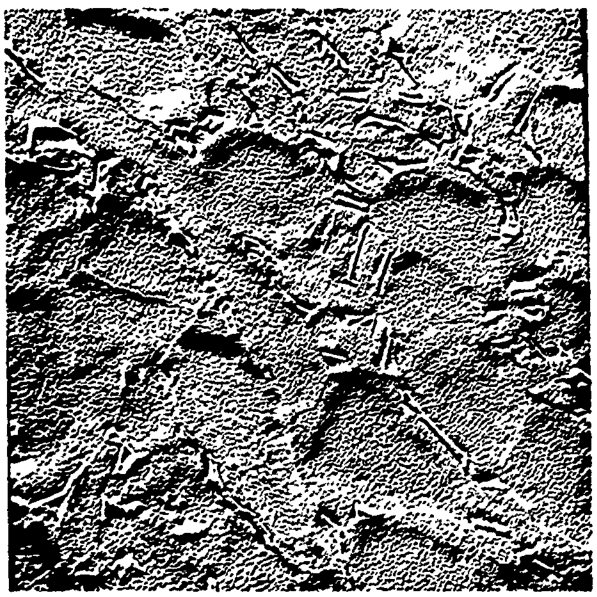


FIGURE 11. (800°F) TENSILE PROPERTIES OF THERMALLY EXPOSED AND CREEP STRAINED Ti-8Al-1Mo-1V

2024-T3 - As-received Condition

5000X



Ti-6Al-4V Annealed As-received Condition

5000X

Ti-8Al-1Mo-1V - Duplex-Annealed As-received Condition

5000X

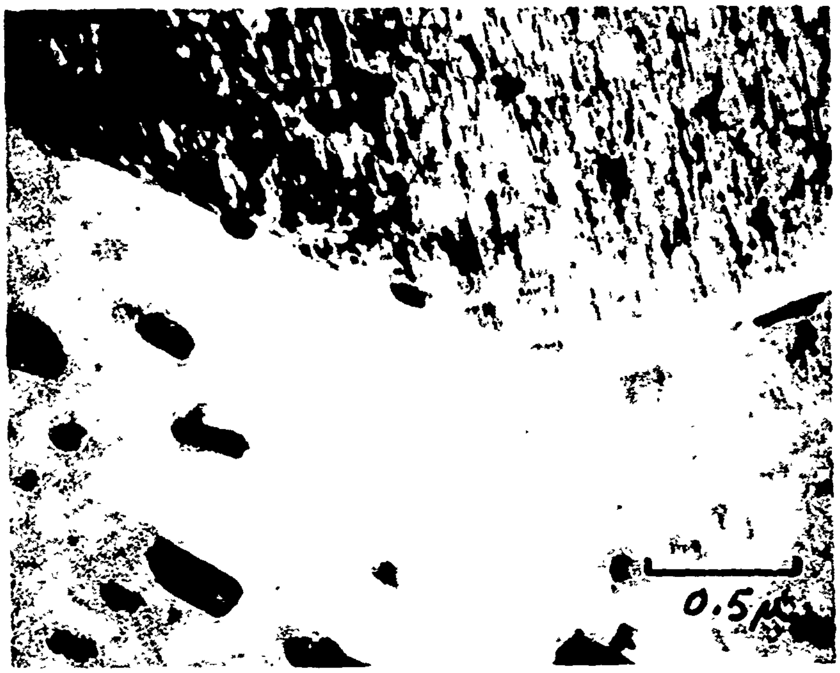
Figure 12 Microstructure of As-Received Materials

Figure 13. Transmission Electron Micrograph of 2024-T3 Aluminum, 50,000X

Figure 14. Transmission Electron Photomicrograph of Equixed α-Ti area in Ti-6Al-4V alloy in As-Received Condition (NOTE: Dislocation tangles at A and B)

Figure 15. Transmission Electron Photomicrograph of Aged β-Ti grain in Ti-6Al-4V Alloy in As-Received Condition.

Figure 16 Transmission Electron Photomicrograph of α-Ti grain in Ti-8Al-1Mo-1V Alloy in As-Received Condition (NOTE: Particles of TiH or TiO and long, partially pinned dislocations [A])



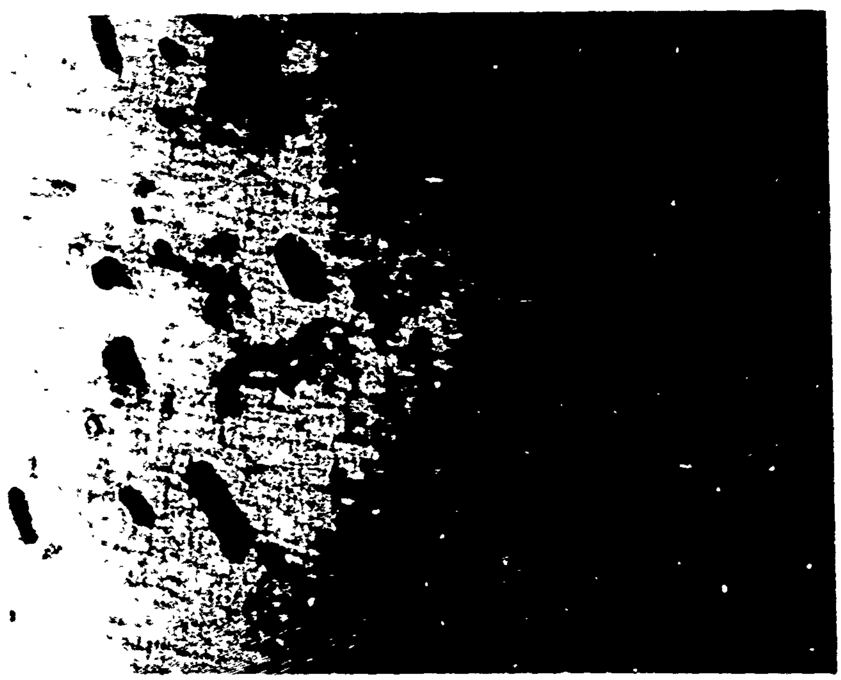


Figure 17. Transmission Electron Micrographs of 2024-T3, Thermally Exposed to 300°F for 50 Hours (NOTE: Formation of θ" Very Fine Platelets)

Figure 18. Transmission Electron Micrograph of 2024-T3, Thermally Exposed to 300°F for 250 Hours (NOTE: Platelet Structure θ')

Figure 19 Transmission Electron Micrograph of 2024-T3, Thermally Exposed 500 Hours at 300°F (NOTE: Platelet Structure θ' and θ - $CuAl_2$)

Figure 20. Transmission Electron Photomicrograph of Ti-8Al-1Mo-1V After Thermal Exposure of 50 Hours at 800°F (NOTE: Grain in center of β-phase, dislocations at [A])

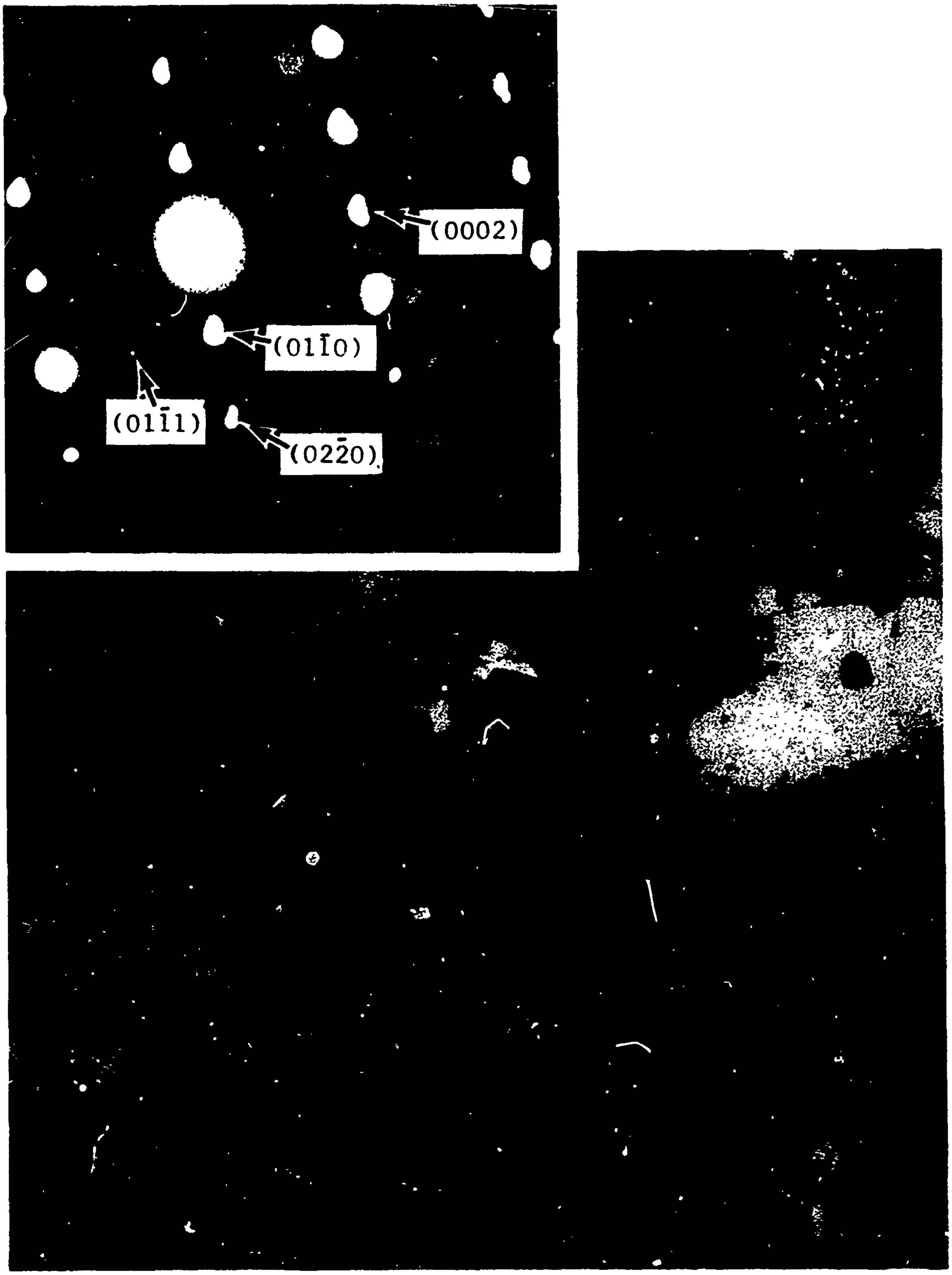


Figure 21. Transmission Electron Photomicrograph of Ti-8Al-1Mo-1V After Thermal Exposure of 500 Hours at 800°F (NOTE: Fine precipitate identified by SAD as α_2 phase; dislocation tangle, [A], polygonization of dislocations at [B])

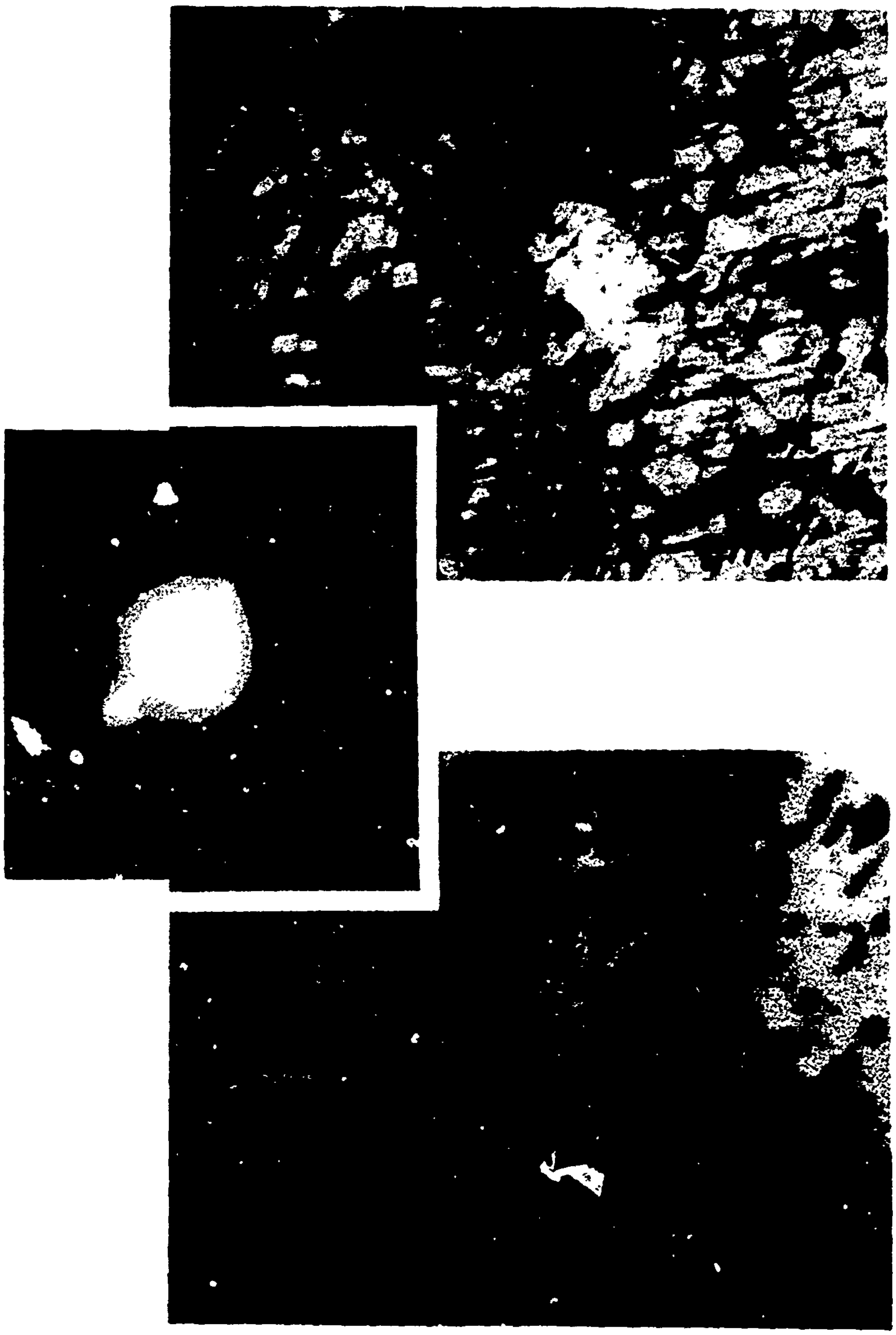


Figure 22. Transmission Electron Micrography of 2024-T3, Creep Strained to 1% Strain at 300°F in 50 Hours (Platelet Structure is θ' or θ' + θ'') (Crystallographic direction normal to the photograph)

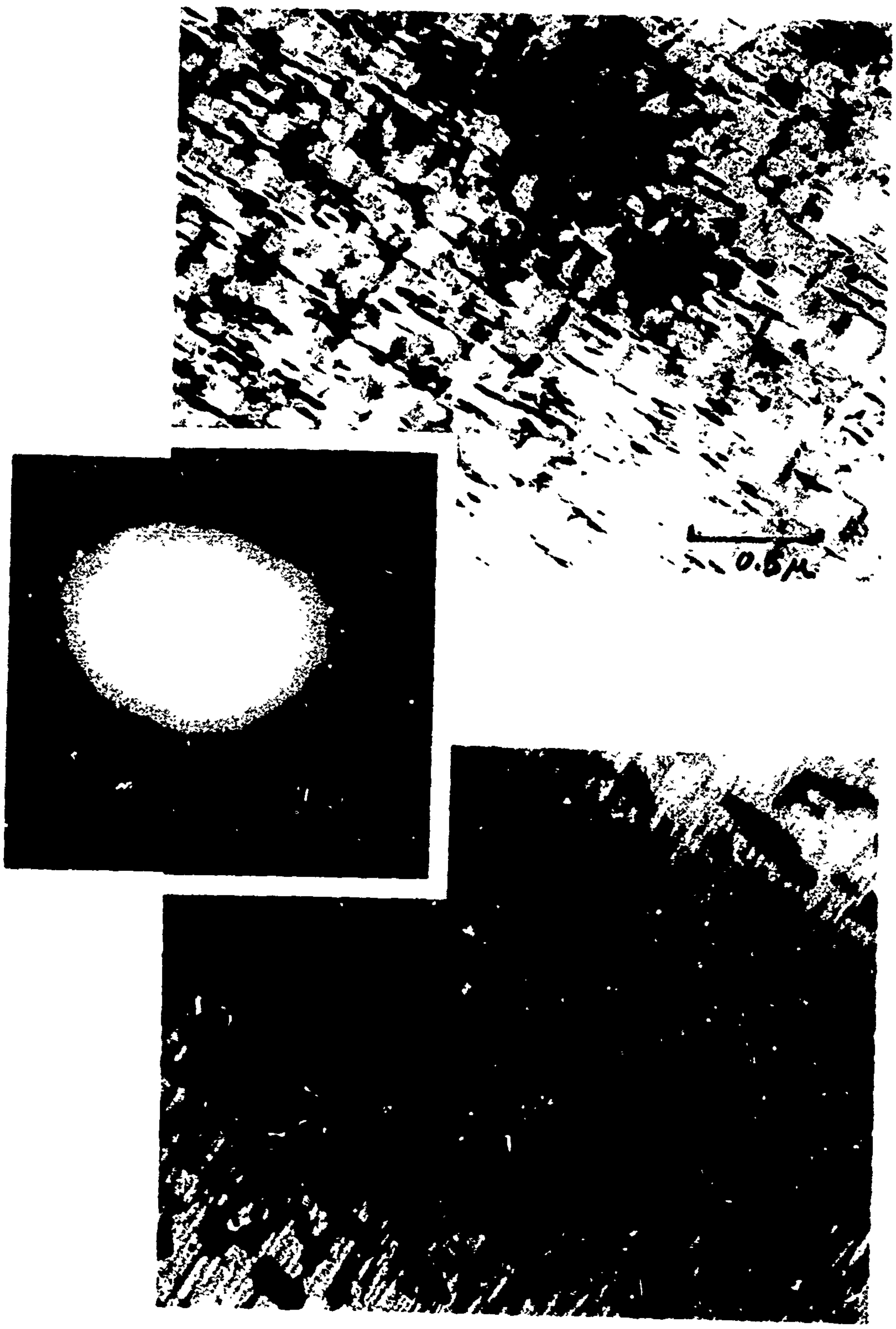


Figure 23 Transmission Electron Micrograph of 2024-T3, Creep Strained to 1% Strain in 500 Hours at 300°F (NOTE: Overaging of θ' is top photo, and high density of θ is lower photo)

FIGURE 24 SUBSTRUCTURE OF Ti-6Al-4V STRAINED TO 1% CREEP STRAIN IN 50 HOURS AT 800F. NOTE SLIP BANDS (A)

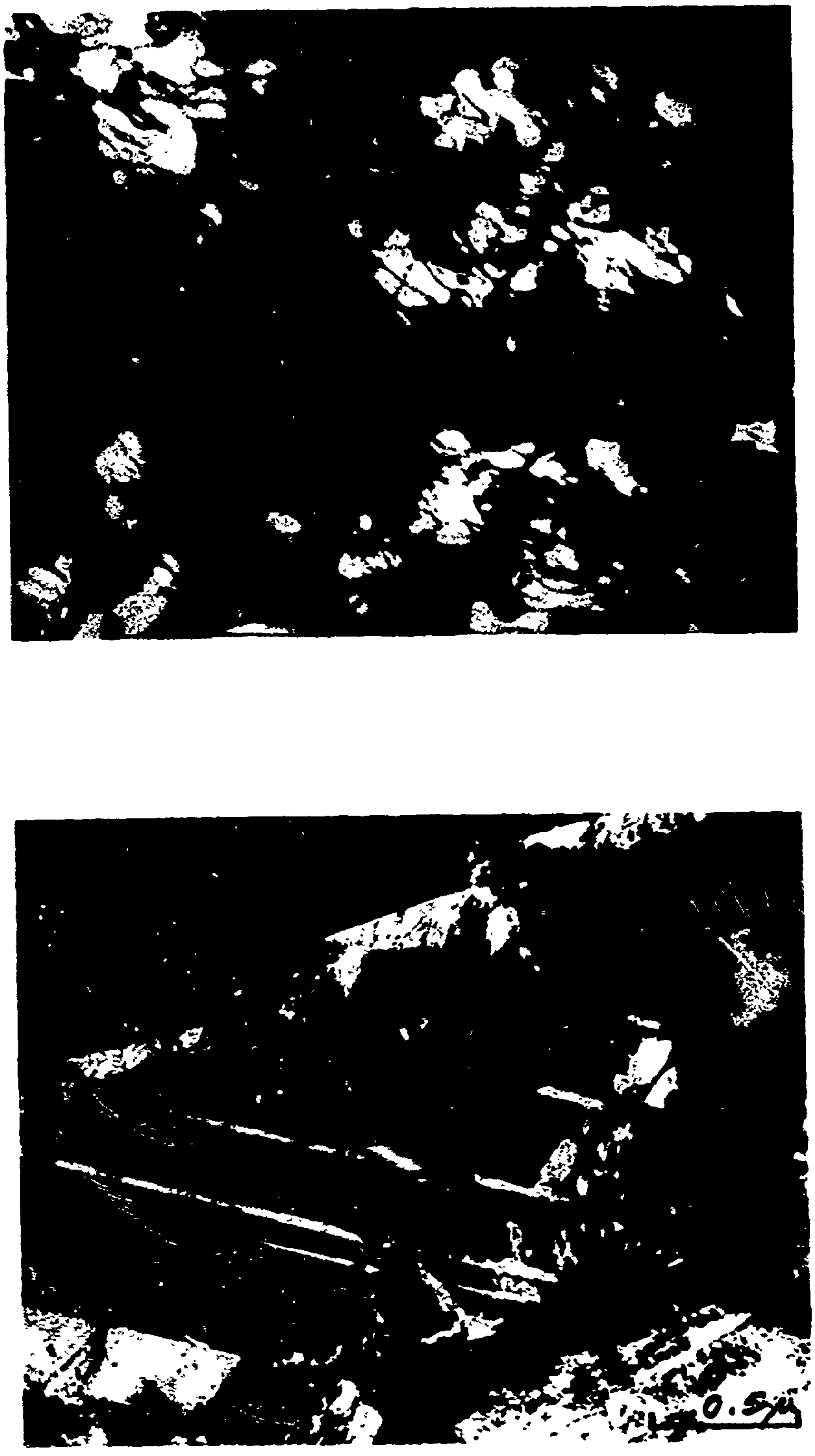
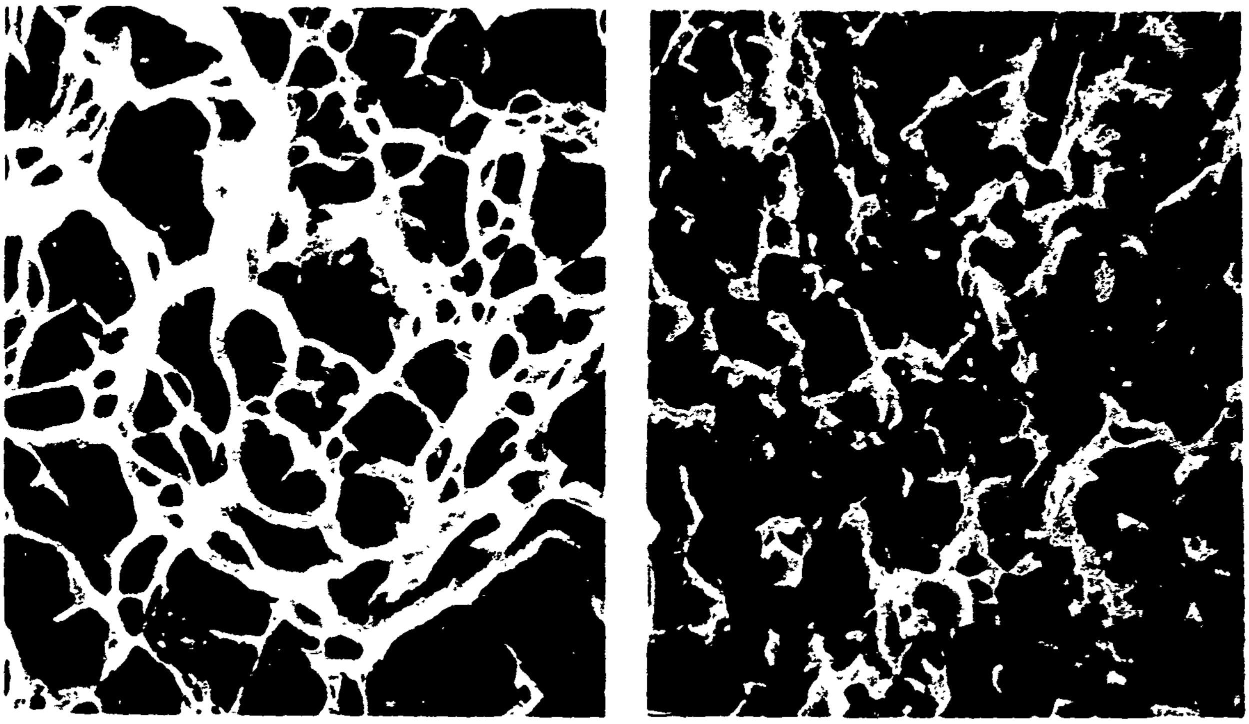


Figure 25 Substructure of Ti-6Al-4V Strained to 1% Creep Strain in 500 Hours at 800°F (NOTE: Slip lines in upper photo and twinning in lower)

FIGURE 26. SUBSTRUCTURE OF Ti-8Al-1Mo-1V STRAINED TO 1% CREEP IN 50 HOURS AT 800F.



1000X

500 Hours Exposure at 800°F Thermal,
RT Tensile Test

1000X

1% Strain in 500 Hours at 800°F,
RT Tensile Test

Figure 27. Comparison of Fracture Surfaces of Ti-8Al-1Mo-1V Specimens

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11 SUPPLEMENTARY NOTES	12 SPONSORING MILITARY ACTIVITY Air Force Materials Laboratory Wright-Patterson Air Force Base Ohio 45433

13 ABSTRACT

This investigation was performed to study the validity of certain criteria used to achieve compression of test time of elevated temperature creep tests. The hypothesis that accumulation of a constant strain under various stresses at a constant temperature results in equivalent damage was evaluated from residual strength level as well as microstructural behavior. Materials selected for this evaluation were Ti-8Al-1Mo-1V in the duplex-annealed condition, Ti-6Al-4V in the annealed condition, and aluminum alloy 2024-T3. Materials were chosen as representative of high strength titanium alloys and aluminum alloys having good elevated-temperature strength. The titanium alloys were creep strained to 1% total strain using three different creep stresses at 800F; the aluminum alloy was investigated using a similar approach at 300F. Detailed microscopic studies were performed to study microstructural changes in terms of the creep rate. Residual strength was correlated with microstructure to determine the validity of the "equivalent damage" approach to test time compression.

It was determined that the hypothesis of a constant strain resulting in equivalent damage was not universally valid. Metallurgical changes as a result of thermal exposure and creep straining resulted in changes in tensile strength behavior. Examination of the microstructure could be directly correlated with mechanical behavior changes.

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Prior Creep Effects						
Creep Data						
Precipitate Structures						
Deformation Phenomena						
Test Time Compression						

END

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